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**EDGEWOOD ARSENAL
TECHNICAL REPORT**

EATR 4085

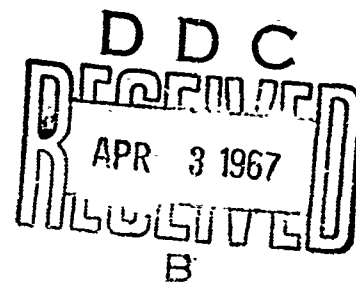
**VACUUM (OR FLUXLESS) BRAZING-GAS
QUENCHING OF 6061 ALUMINUM ALLOY**

by

M. M. Schwartz
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Francis B. Gurtner and Paul K. Shutt, Jr.
Technical Support Directorate

March 1967



**Pre-Production Evaluation Division
Technical Support Directorate
EDGEWOOD ARSENAL
EDGEWOOD ARSENAL, MARYLAND 21010**

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FOREWORD

The investigation was started in January 1965; the initial phase has been completed, and advance work is progressing.

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DIGEST

The purpose of this investigation was to provide a process or method for producing helium leak-tight containers under controlled processing and therefore with a high level of compatibility.

The concept evolved around combining vacuum brazing and gas quenching into one operation. The parts were quenched in the furnace after brazing or solution treating.

The advance into the field of vacuum brazing-gas quenching has provided a definite advance in the field of manufacturing for high or low volumes of items.

As a result of the investigation, the following conclusions were drawn:

1. Vacuum brazing-gas quenching is feasible for production quantities of complex and simple items.
2. The application of such a process is not limited to 6061 aluminum, but can be extended to ferrous and nonferrous alloys.
3. The future for a process such as vacuum brazing-gas quenching is unlimited at the present time.

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VACUUM (OR FLUXLESS) BRAZING-GAS QUENCHING OF 6061 ALUMINUM ALLOY

I. INTRODUCTION.

As more and more new metals are being developed, such as the super-alloy and refractory-type metals, the potentials and capabilities of existing joining techniques are being challenged by present-day technology.

The culprit in most joining operations is contamination in one form or another. With the current interest in vacuum technology increasing, the authors decided to use vacuum brazing in an effort to control or eliminate the critical oxide contamination present with other joining techniques. Vacuum brazing eliminates the need for brazing flux, thereby lowering costs and excluding poor joint quality and corrosive damage, and eliminates the need for organic adhesives, which are often unsuitable at cryogenic or moderately high temperatures. There seems to be no reason why aluminum could not be vacuum-brazed and why the process could not be used (1) in brazing radiators and heat exchangers for aircraft and missiles, wave-guide fittings, and nuclear work; (2) in instances in which residual flux functionally poisons parts such as condensers and evaporators; and (3) in instances in which service requirements are too severe for organic adhesives or other joining materials and processes.

Gas quenching was tried because the authors wanted to maintain a high quality level in joining with respect to cleanliness, corrosion resistance, and compatibility. It was desired to eliminate water as a quenching medium and combine vacuum brazing and gas quenching into a single productive controlled function. Using this process, the postcleaning of assemblies is completely eliminated, and aluminum or steel tooling can be used instead of the more expensive stainless steel and nickel alloy tools.

II. EXPERIMENTATION.

A. Alloy Selection.

The aluminum alloys considered for vacuum brazing are those currently being brazed; namely, 2002, 3003, 6061, 2219, and 7005. The braze alloy was No. 718 (88% aluminum, 12% silicon), which is used in most brazing work today and usually in wire or foil form.

The initial tests were on lap joints and were conducted using nickel and silver plate for wetting. The results of these tests are described in table I. All failures were next to the lap joint. It was decided to examine the possibility of brazing without any coating.

Table I. Results of Physical-Stress Tests on 0.125-in.
6061 Lap-Joint-Brazed Strips

Strip No.	Maximum load	Strip No.	Maximum load
	lb		lb
Ag-1	1,100	P-1	1,260
Ag-2	1,290	P-2	1,250
Ag-3	1,210	P-3	1,270
Ag-4	1,210	P-4	1,235*
Ag-5	1,140	P-5	1,255*
Ni-1	1,110	169-1	1,090
Ni-2	1,230	169-2	1,265
Ni-3	1,175	169-3	1,470**
Ni-4	1,105	169-4	1,185
Ni-5	1,050		

* Weld showed on Nos. P-4 and P-5; in all others, parent metal failed at edge of weld.

** Failed in grip at 1,365 lb; reran broken section; final break at 1,470 lb.

B. Cleaning and Initial Test Results.

The initial step in producing good brazed joints is to clean the aluminum surface. The cleanliness of the surface to be brazed is quite critical, and it was determined that brazing must take place within 12 hr after cleaning. The steps in cleaning are as follows:

1. Vapor degrease
2. Alkaline clean
3. Water rinse
4. Nitric acid dip
5. Nitric-hydrofluoric acid dip
6. Water rinse
7. Oven dry at 230°F

In addition, joint design, assembly tolerances, and general brazing practices that have been in use with either dip or furnace brazing, in which a flux was used to promote metal flow, were also adequate for vacuum brazing. The brazing time was 1 to 2 min, although allowance was made for the special heat-transfer problems inherent in a vacuum and for the capacity of the vacuum equipment to pump down to the proper limits. Brazing temperatures were 1,080° (minimum) to 1,100°F (maximum), with a $\pm 10^\circ\text{F}$ nominal tolerance range and a vacuum of 1×10^{-6} torr. Figure A-1* shows the interior of the vacuum furnace used. Initial tests were conducted on 0.040-in.-gauge 6061 sheet material, which caused failures in the base metal; therefore, to achieve lap shear results, 0.125-in.-gauge material was used. The test results are shown in table II. In addition, several dip-brazed and base-metal specimens were tested for comparison.

Table II. Comparison of Vacuum- and Dip-Brazing of
0.125-in. 6061 Aluminum Sheet

Type of braz	Dimensions	Area	Ultimate load	Strength stress
	in.	sq in.	lb	psi
Vacuum	0.125 x 0.75	0.0937	880	9,400
Vacuum	0.125 x 0.75	0.0937	930	9,800
Vacuum	0.125 x 0.75	0.0937	955	10,200
Dip	0.125 x 0.75	0.0937	1,030	11,000
Dip	0.125 x 1.0	0.125	1,218	9,750
Dip	0.125 x 1.0	0.125	1,147	9,180
Vacuum	0.125 x 1.0	0.125	1,260	10,080
Vacuum	0.125 x 1.0	0.125	1,155	9,230
Base metal	0.125 x 1.0	0.125	1,580	12,600

C. Application.

With the encouraging results gained from test specimens, the process was applied to brazing actual hardware. Water was eliminated as a quenching medium because of its incompatibility with various reagents; an inert gas was substituted in order to achieve a T-4 condition in the base metal after brazing.

The first series of tests of time versus temperature was conducted using helium, argon, and liquid nitrogen. The test results showed helium to be the best. It was possible to lower the temperatures of the furnace and the part from the braze temperatures much more rapidly with helium than with any other gas. The dew point of helium and argon in all tests was -76°F minimum. All brazed and quenched bomblet canisters met the leak-rate criterion of 1×10^{-6} cc/sec for 15 sec (figure A-2).

The brazing clearance used was an 0.002-in.-interference fit. The maximum joint clearance was not determined.

* All figures, A-1 through A-23, are in the appendix.

The braze and heat-treat cycle consisted of:

1. Heat to 980°F and hold 30 min.
2. Raise temperature to 1,080°F and hold 30 sec.
3. Introduce helium and cool.

III. DISCUSSION.

A. Gas Quenching.

The use of gas quenching for the manufacture of bomblet canisters is increasing because of the necessity to maintain a high quality level. The quality level is based upon three processing standards - cleanliness, corrosion resistance, and compatibility. When a bomblet is quenched by the common water method, it is impossible to remove completely the water and other compounds formed upon quenching. These residuals are the main cause of defectiveness in manufactured bomblet canisters.

The cleanliness level that is produced as a result of gas quenching does not, as of this writing, have a classification. Classifications for cleaning levels are going to be established in the near future. By cleanliness level, we are implying that laboratory levels of cleanliness are being used under production conditions. Corrosion in this investigation refers to an acid build-up and subsequent deterioration of the 6061-T6 aluminum alloy and a total loss of the part that has been under surveillance. Compatibility is the ability to store any compound inside the container under all types of conditions without any resultant reactions.

Figures A-3 and A-4 show two types of bomblet canisters that cannot be quenched by the water method. The maximum hole diameter for the passage of water is only 0.125 in.; it is in turn restricted by a deep well, which is only 0.060 in. from the 0.125-in. hole.

The gases first considered were helium, argon, and nitrogen, because of their relatively inert classification and lack of residual reaction with 6061 aluminum. Any oxidation of the radiant heating element would be kept to a minimum, as quenching would be accomplished in the furnace. The thermal conductivity of the three proposed gases is:

1. Helium - 3.32×10^{-4} Cal/sq cm/cm/sec/°C
2. Argon - 0.406×10^{-4} Cal/sq cm/cm/sec/°C
3. Nitrogen - 0.60×10^{-4} Cal/sq cm/cm/sec/°C

Of the three gases used for quenching from either the brazing temperature of 1,080° to 1,105°F or the solution-treating temperature of 980°F, helium has the best quench rate (figures A-5 to A-7). The curves illustrate

the rates of quenching for both the furnace and the part. The most important curve is the one for the part. The first results from gas quenching were thought to be very good when the quenching rate was compared with the furnace-temperature charts, but the physical properties and hardness values depict a different story. The initial hardness values* for 6061 aluminum quenched by gases (utilizing 3/8-in. copper tubing to transfer the gas) are:

Argon

R_H 74

R_H 77 (15 days later)

Helium

R_H 75.5

R_H 78.5 (15 days later)

The tensile strength was in the range of 23,000 to 24,000 psi. To facilitate better quenching, a 1-in. line was installed in place of the 3/8-in. line, and to obtain more data, thermocouples were attached to the parts. The change in the quenching medium and in the method of recording produced immediate results. The hardness values increased to a range of R_H 79 to 80 for helium and R_H 78 to 79 for argon. According to The Metals Handbook, 8th edition, the hardness for 6061 in T-4 condition is R_H 80 to 102, with a minimum tensile value of 35,000 psi. The values given in table III show the reduced values. The designations refer to the following conditions:

1. MB1-1 - furnace-cooled
2. MB2-1 - helium-quenched
3. MB3-1 - helium-quenched
4. MB4-1 - argon-quenched
5. EA - water-quenched (standard)

Table III. Comparison of Tensile Strength
of Brazed Strips as Received

Designation	Width	Thickness	Area	Load	Tensile strength	Elongation
	in.	in.	sq in.	lb	psi	%
MB1-1	0.5035	0.042	0.02115	435	20,565	21.5
MB2-1	0.503	0.043	0.02163	550	25,425	21.0
MB3-1	0.5035	0.042	0.02115	545	25,770	22.0
MB4-1	0.503	0.042	0.02113	490	23,190	20.0
EA-1	0.504	0.042	0.02117	795	37,555	22.0
EA-2	0.5055	0.0425	0.02148	795	37,010	22.0
EA-3	0.5025	0.042	0.02111	790	37,425	24.0

* Rockwell H.

The hardness values (both R_H and R_{15T}) for samples MB1, MB2, MB3, MB4, and EA are given in table IV. The original goal of R_H 80 had been achieved (helium), even though the tensile strength did not reach 35,000 psi.

Table IV. Hardness Values of Gas-Quenched Brazen Strips

Designation	Hardness								
	Rockwell H						15T		
	End 1			End 2					
MB1-1	68.5	69.5	69.5	70.0	71.0	70.0	49.5	46.0	48.0
MB1-2	70.0	72.5	70.5						
MB1-3	70.0	71.0	72.0						
MB2-1	81.5	81.0	82.5	96.0	97.0	95.5	58.0	57.5	58.0
MB2-2	83.0	83.0	82.5						
MB2-3	81.0	80.5	81.0						
MB3-1	82.5	82.0	81.0				61.0	57.0	57.0
MB3-2	81.0	81.0	81.0						
MB3-3	80.0	80.5	81.0						
MB4-1	77.5	76.0	76.0				56.0	54.0	53.5
MB4-2	77.0	78.0	78.0						
MB4-3	77.0	74.5	76.5						
EA-1	94.0	94.0	95.0				69.0	72.5	72.0
EA-2	95.5	96.5	96.0						
EA-3	95.0	95.5	97.0						
EA-4	95.0	95.5	96.0						
EA-5	96.5	96.5	96.5						
							73.0	73.5	74.0

B. Microstructures.

A survey of literature on the microstructure of aluminum alloys produced little information on isothermal curves and less on the T-6 condition, which is the final condition required for structural loading. With the considerable lack of information, two approaches were undertaken to gain more information: the development of isothermal curves and the determination of the metallographic structure of 6061 in the T-6 condition. The 6061 aluminum alloy, upon furnace cooling and gas quenching with argon and helium, was examined to provide an index for classifying the precipitate percentage. A percentage factor was related to each method of quenching so that an aging cycle could be developed proportional to the best conditions.

Operations to date have not permitted a thorough study of the structures and of their relationship to processing and heat-treating conditions. Previous conditioning of the material, which is described later, has a direct bearing on the final structure. In addition, 6061 aluminum alloy has a memory that cannot be removed through reheating.

The effects of air, argon, helium, and nitrogen quenching are illustrated by the microstructures in figures A-8 through A-13 (the aluminum was etched with sodium hydroxide).

Figure A-8 depicts the structure produced after the sample was brazed at 1,105°F and then cooled in the vacuum furnace. The same sample was used to determine and evaluate the effects of the gases and the heat-treating cycle upon the canisters.

Figures A-9 through A-13 show the structures produced by brazing, heat-treatment and quenching using varying methods.

Work is now underway for gas quenching bombulet canisters through several varying approaches. At present, retorts to contain the canisters and in which the gas-quenching operation can be applied are being investigated. Several interesting preliminary approaches are being tried: utilization of oxidizing gases and oxidizing and inert gases in combination; elimination of pressure gages; and altering the capacity and location of exhausts in order to attain a quench delay time of 2 min from a temperature of 980° to 0°F.

C. Solution Cycles.

To try for a greater hardness in the T-4 condition, other solution-treating cycles were investigated, for instance, a double cycle.

1. First Cycle

- a. Step One: Anneal at 800°F for 2.5 hr.
- b. Step Two: Increase temperature for solution-treating to 960° to 980°F and hold for 45 min.
- c. Step Three: Increase furnace temperature to 1,080°F and hold for brazing.
- d. Step Four: Gas-quench from brazing temperature.

2. Second Cycle

- a. Step One: Increase furnace temperature to 960° to 980°F and hold for 45 min.

b. Step Two: Increase furnace temperature to 1,080°F and hold for brazing.

c. Step Three: Gas-quench from brazing temperature.

In the quenching operation for the first and second cycles, three large cylinders were manifolded together and pressurized for immediate release into the furnace. The full volume from all three cylinders was expended on each furnace run.

Cycle No. 1 did not produce the hardnesses of R_H 85 to 90 that would have been anticipated by the additional time for annealing. The hardness value was lower using this cycle, and, therefore, the furnace runs were returned to the short cycle, No. 2.

To prove the feasibility of brazing and gas-quenching bomblet canisters under production conditions, several furnace runs were established per part volume. The volume of parts per run encompassed 25 to 200 units per furnace load without any relative loss in hardness or ultimate strength value. Figure A-14 shows the low-cost tooling for vacuum brazing-gas quenching 200 bomblet canisters at one time; a high or low volume was imposed for a continuous course of performances.

D. Tolerances.

In the day-to-day brazing controls, tolerances are expressed as loose, snug, or interferent, but rarely is a close control established for prepilot production. Several trials were made with fits between loose and snug (no interference) with good braze results, but varying results as to meeting the helium leak criterion of 1×10^{-6} cc/sec for 15 sec. After several trials and a low yield, 80% per furnace run, corrections were made on one of the details that provided an interference tolerance of 0.002 to 0.004 in. between the mating parts that formed the lap joint of 1/8 in. The conformance to the tolerance of 0.002 to 0.004 in. produced a reliability of 97.6% from the brazing-quenching operation through a 100% 1×10^{-6} cc/sec helium leak test quality level.

E. Aging.

The standard aging cycle of 6 to 8 hr at 350°F was tried to determine the reaction of the bomblet canisters with a hardness of R_H 81. The results were poor, as almost no change took place.

At this point, several aging cycles were proposed, and four were tried for evaluation: 300° to 320°F for 67 hr; 300° to 320°F for 39 hr; 350° $\pm 10^\circ$ F for 8 hr; and 350° $\pm 10^\circ$ F for 6 hr, 190° $\pm 10^\circ$ F for 48 hr.

The results from three of the four aging cycles were encouraging for the use of gas-quenched units under actual test conditions.

1. Aging Cycle No. 1.
 - a. Hardness as quenched in helium - R_H 81 to 82
 - b. 300° to $320^\circ F$ for 67 hr - R_H 88 to 96
 - c. Ultimate tensile range - 31,043 to 34,360 psi
 - d. Elongation, 13% to 13.5% (the low elongation corresponds to the low tensile value)

2. Aging Cycle No. 2.

- a. Hardness as quenched in helium - R_H 81 to 82
- b. 300° to $320^\circ F$ for 39 hr - R_H 97 to 99
- c. Ultimate tensile range - 34,000 to 35,000 psi

3. Aging Cycle No. 3.

No change.

4. Aging Cycle No. 4.

- a. Hardness as quenched in helium - R_H 81 to 82
- b. $350^\circ \pm 10^\circ F$ for 6 hr, $190^\circ \pm 10^\circ F$ for 48 hr - R_H 93 to 98
- c. Ultimate tensile range, 33,333 to 35,000 psi
- d. Elongation, 9.5% to 12.5% (the low elongation corresponds to the low tensile value)

Figure A-15 illustrates the class of microstructures that has been persistent throughout the investigation of gas quenching and aging. The micro-constituents are distributed in a dispersion pattern and indicate a dispersion hardening effect upon the 6061 aluminum alloy (etched in sodium hydroxide). Further studies are underway to analyze and study the type of precipitate and its tentative mechanics of formation.

F. Quality Control.

The areas of investigation did not end with the development of an aging cycle, because the isothermal curve on the duplex aging cycle had been overlooked as to the effects. To initiate a proper duplex aging cycle, the uppermost temperature has to be lowered within 22 to 23 min and sloped to a temperature beyond the second cycle. Figure A-16 illustrates a typical duplex cycle: $350^\circ \pm 10^\circ F$ for 6 hr and $190^\circ \pm 10^\circ F$ for 48 hr. The cooling from $350^\circ F$

is accomplished by venting the furnace, resetting the temperature, and allowing a thorough circulation throughout the volume of material. Deviations from the temperature slope rates have resulted in material returning to a dead-soft condition.

The quality level of the gas is very critical, as a dew point of -85°F or better is a basic prerequisite. Beyond the dew-point stage, filtration equipment is now being installed and qualified for what compounds are being removed from standard grades of welding gas. Filtration has been successful in removing particles larger than 1μ (micron) by measuring the change in volume over a period of time. Qualitative and quantitative analyses have not yet been performed on the before and after results of filtration.

Measuring the quality level of the braze by nondestructive means has presently been reduced to using the helium leak test only. Figures A-17 to A-19 illustrate radiographs of vacuum brazed-gas quenched bomblet canisters. The radiographs are of two different densities. Figures A-18 and A-19 are of a similar density, and all but No. 5 passed the leak test.

Sectioning of those bomblet canisters that indicate filler metal not fully consumed showed that they still had a complete braze across the $1/8$ -in. joint.

G. Future Applications and Investigations.

The future for the vacuum brazing-gas quenching process is very promising and will extend to items of varying sizes and complexity such as fuel tanks and rocket warheads, depicted in figures A-20 and A-21. In figures A-22 and A-23 are some other typical applications of vacuum aluminum brazing.

Some immediate advantages are:

1. Brazing flux is not used, and this provides a significant cost reduction compared to conventional practices.
2. No entrapped flux residues; thereby, poor joint quality and corrosive damage, which has often resulted in defective brazed aluminum assemblies, are eliminated.
3. Postcleaning of assemblies is completely eliminated.
4. Aluminum or steel tooling can be used instead of the more expensive stainless steel and nickel alloy tools.

In the commercial field, the next possible applications include radiators for automotive and air conditioning services in addition to various types of thermal conditioning sandwich panels and heat exchangers.

Further investigation should be conducted in the following areas:

1. Volume of filler metal versus joint tolerance and length.
2. Improve joint designs.
3. Evaluate other braze filler alloys.
4. Develop better braze alloys for corrosion resistance and leak-tight requirements.
5. Investigate gases as to their effects upon quenching rate, quench rate versus volume.
6. Evaluate nonbrazable alloys (7075, 2014) and the joining of dissimilar combinations such as 6061 to 2219 and aluminum alloys to titanium, etc.

IV. CONCLUSIONS.

It is concluded that:

1. Vacuum brazing-gas quenching is quite feasible for production quantities of complex and simple items.
2. The application of such a process is not limited to 6061 aluminum alone, but can be extended to ferrous and nonferrous alloys.
3. The future for a process such as vacuum brazing-gas quenching is unlimited at the present time.

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APPENDIX

FIGURES

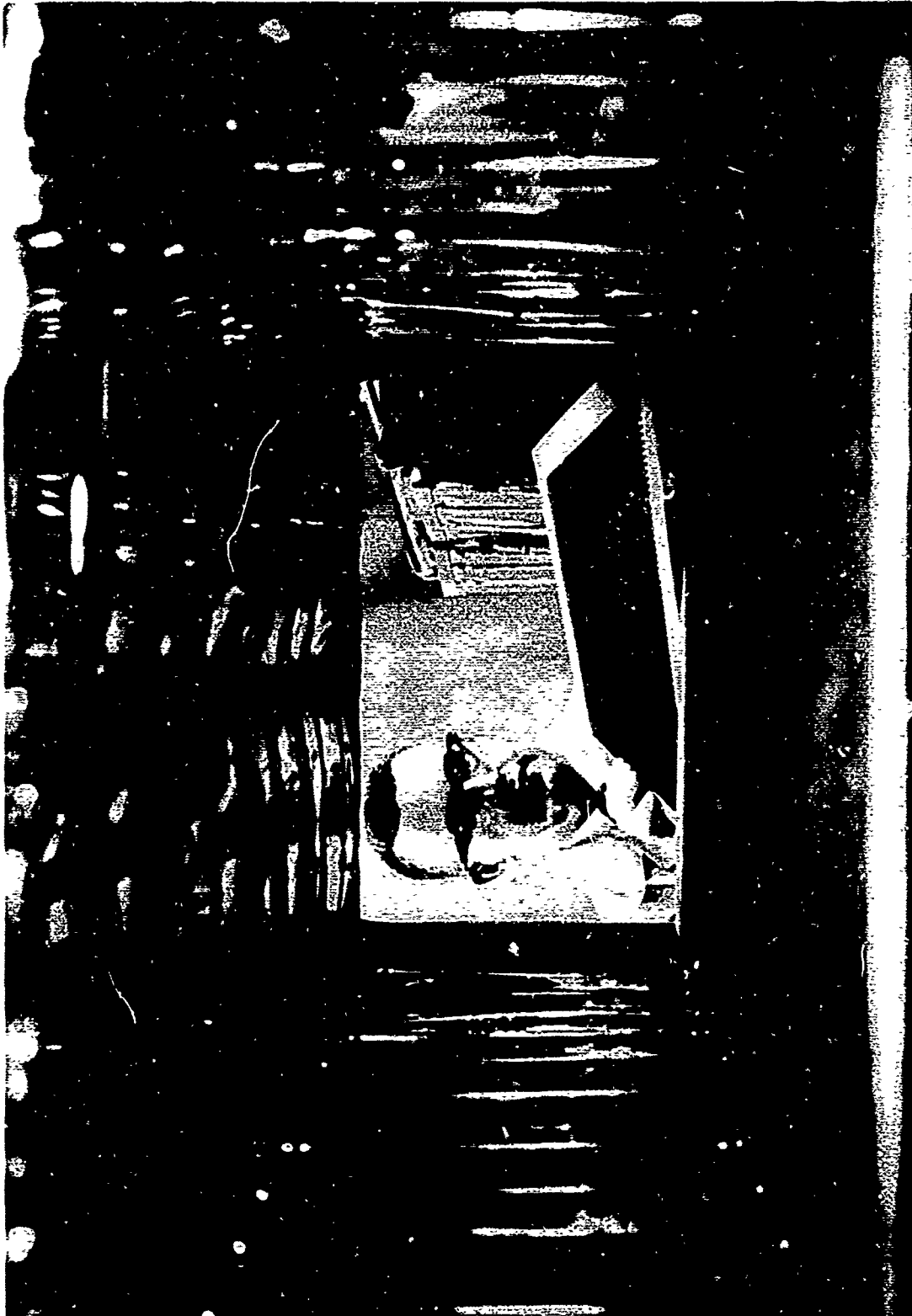


Figure A-1. View of Interior of Vacuum Furnace Utilized for Most Investigations

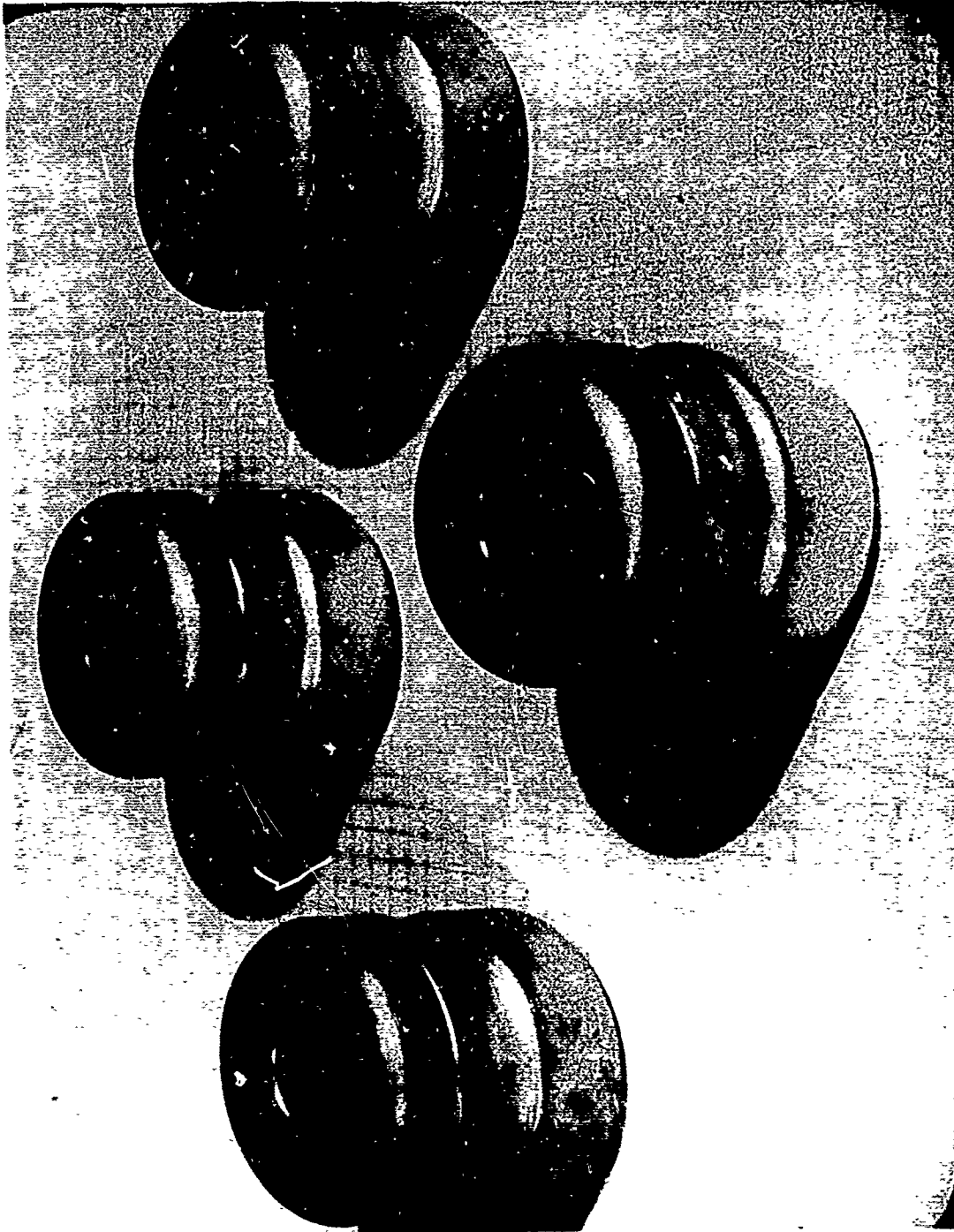


Figure A-2. Vacuum-Brazed, Gas-Quenched Bomblet Canister That Met Helium Leak Criterion of 1×10^{-6} cc/sec for 15 sec

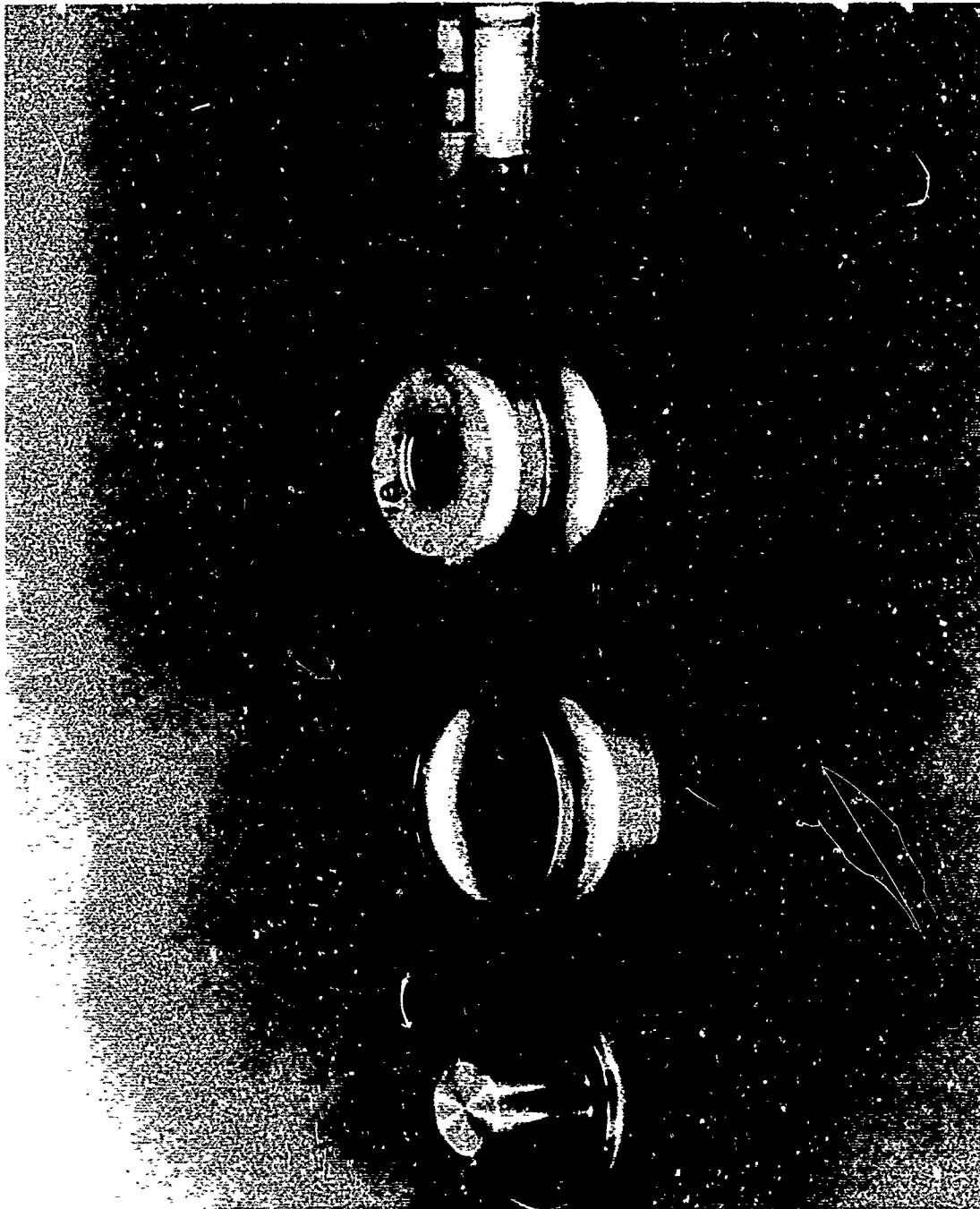


Figure A-3. Vacuum-Brazed, Gas-Quenched Bomblet Canister and Components

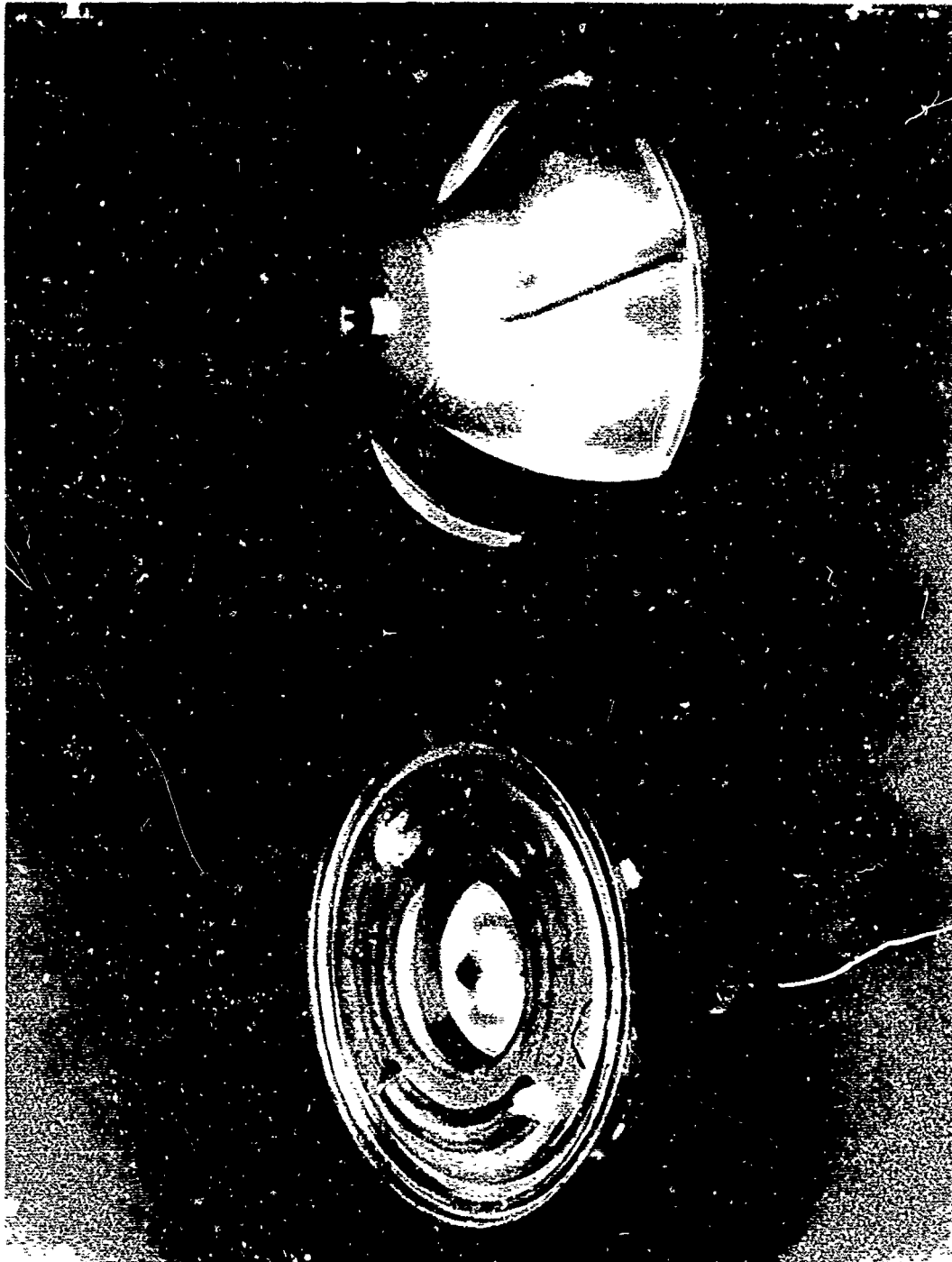


Figure A-4. Vacuum-Brazed, Gas-Quenched Bombulet Canister of Different Configuration

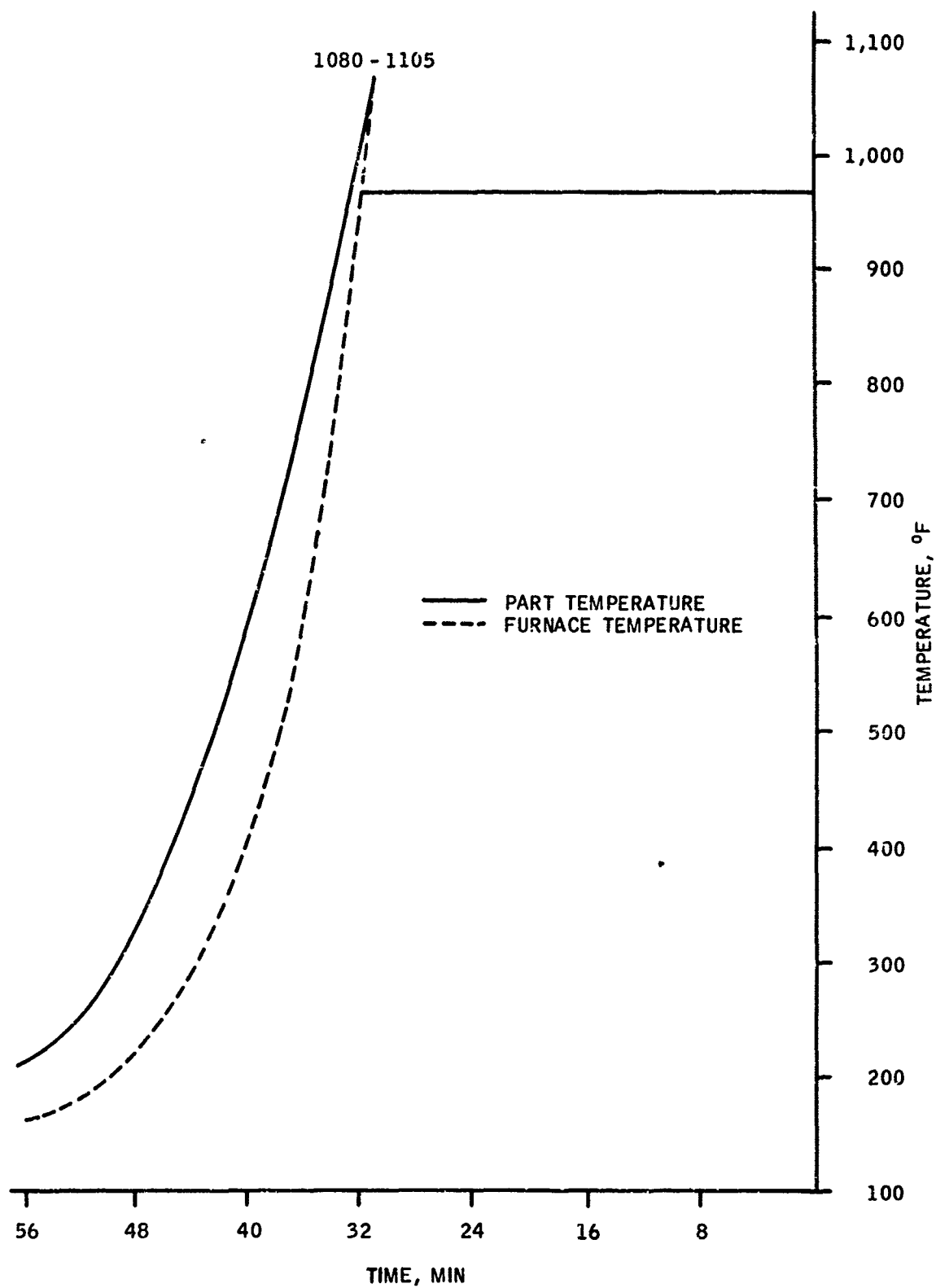


Figure A-5. Quenching Rates for Both Furnace and Part Using Helium Presolution
(Solution-treated at 980°F for 45 min; brazed at 1,080° to 1,105°F)

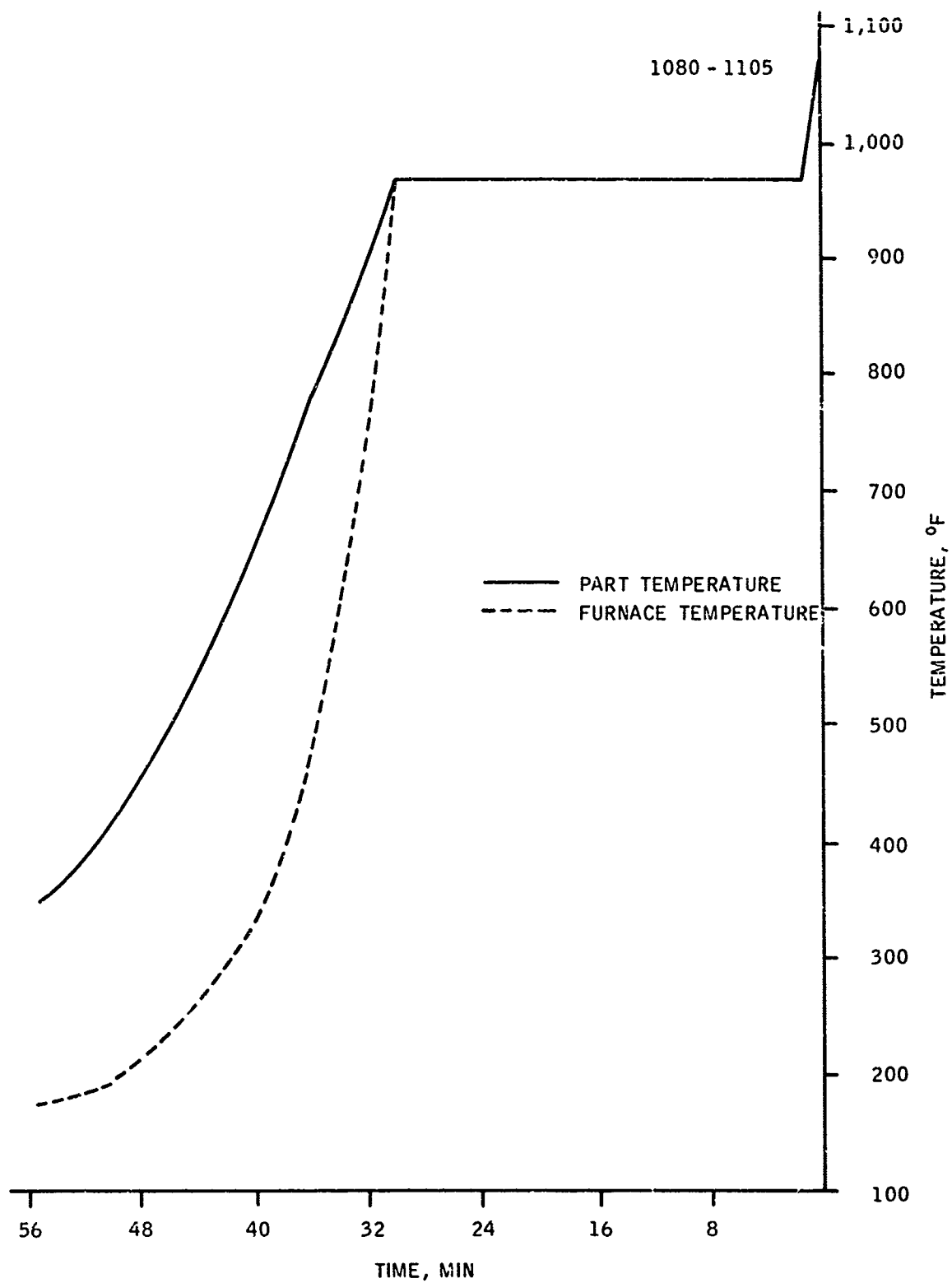


Figure A-6. Quenching Rates for Both Furnace and Part Using Argon Postsolution
(Solution-treated at 980°F for 45 min; brazed at 1,080° to 1,105°F)

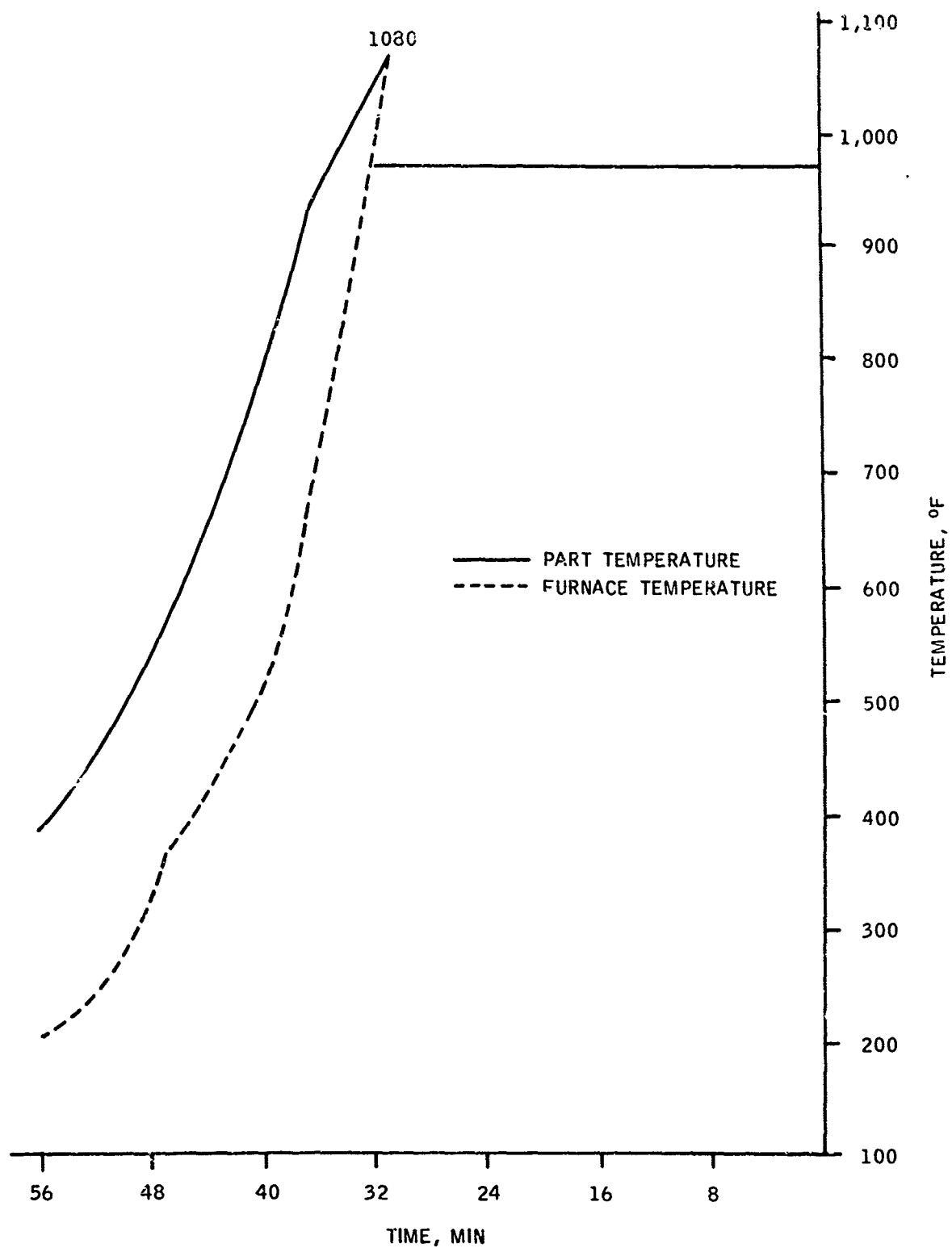


Figure A-7. Quenching Rates for Both Furnace and Part Using Argon Presolution
(Solution-treated at 980°F for 45 min; brazed at 1,080° to 1,105°F)

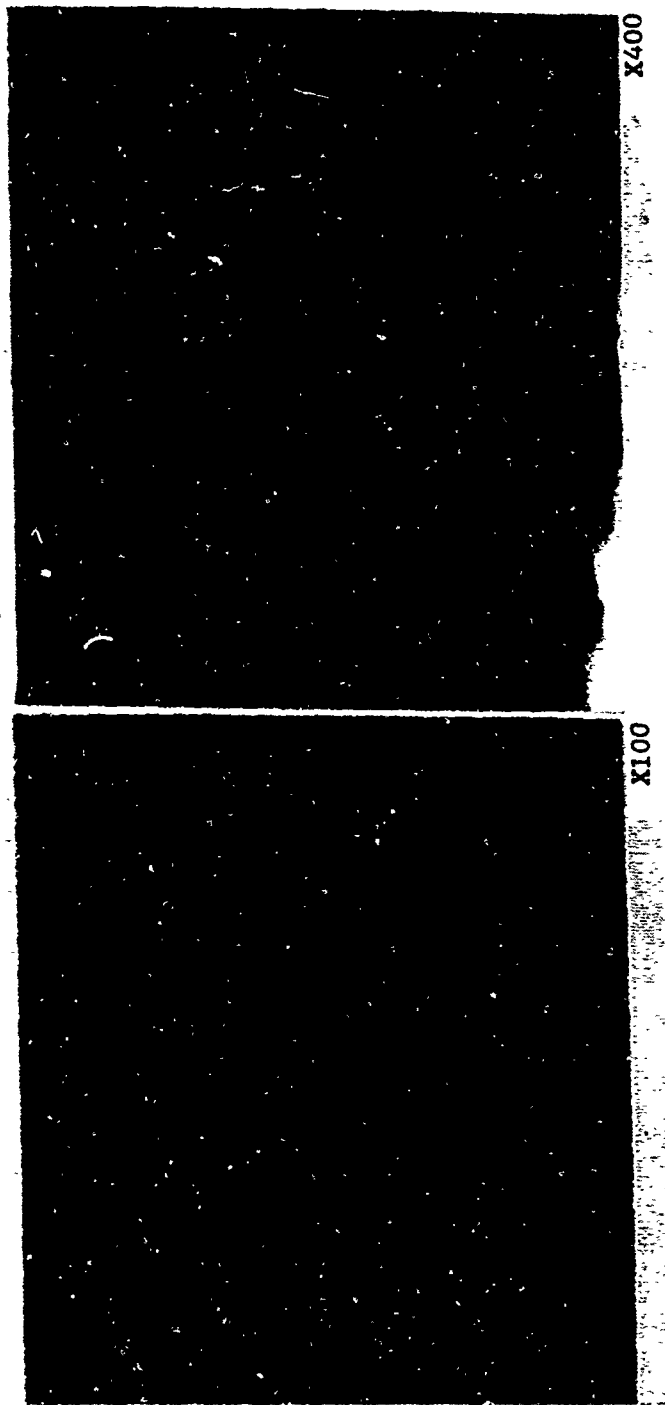


Figure A-8. Microstructures of 6061 Aluminum Brazed at 1,105°F and Furnace-Cooled



Figure A-9. Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Helium

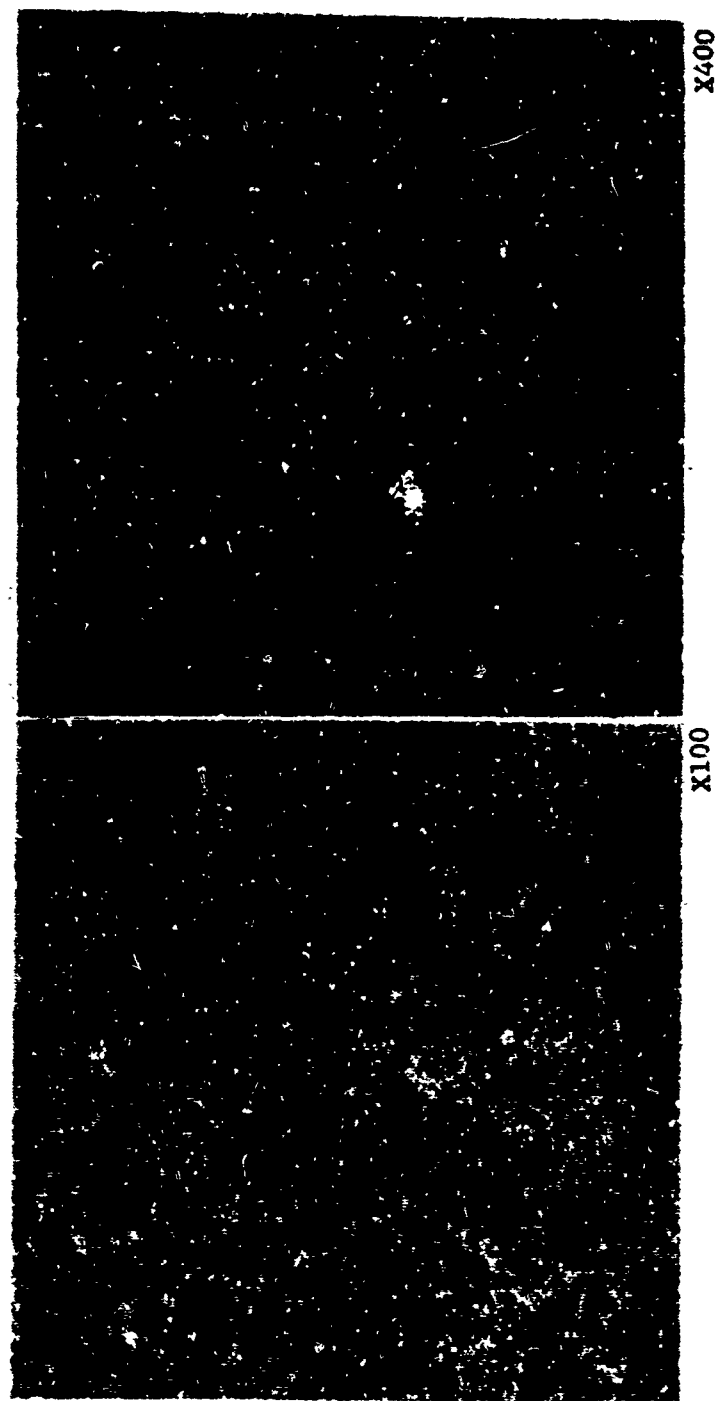
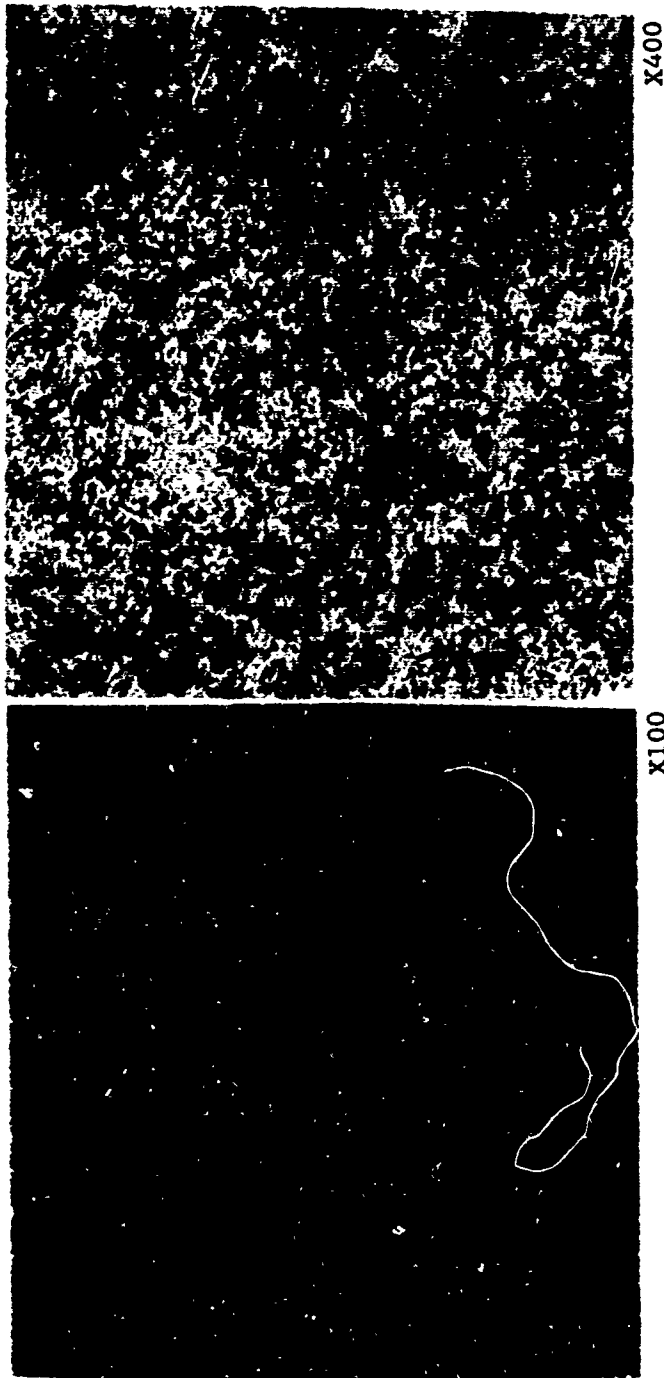


Figure A-10. Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Argon



X400

X100

Figure A-11. Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Helium

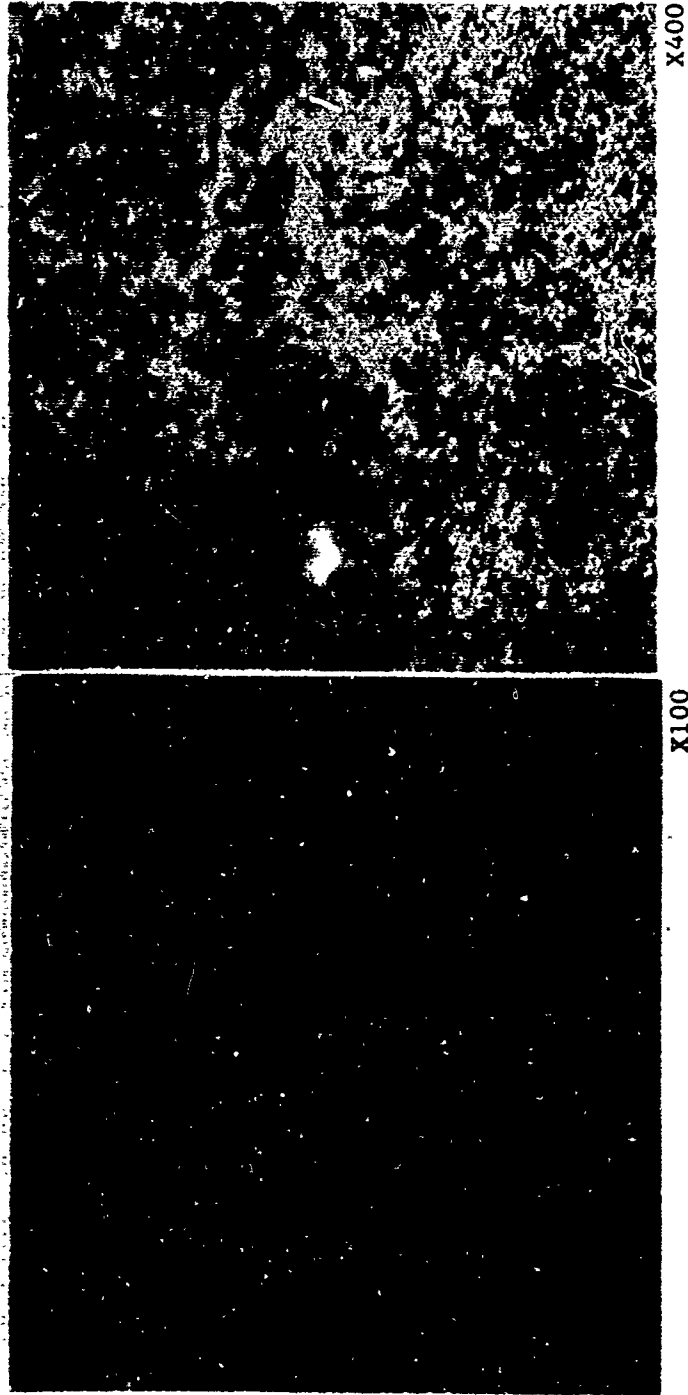


Figure A-12. Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, Brazed at 1,080° to 1,105°F, and Quenched in Nitrogen

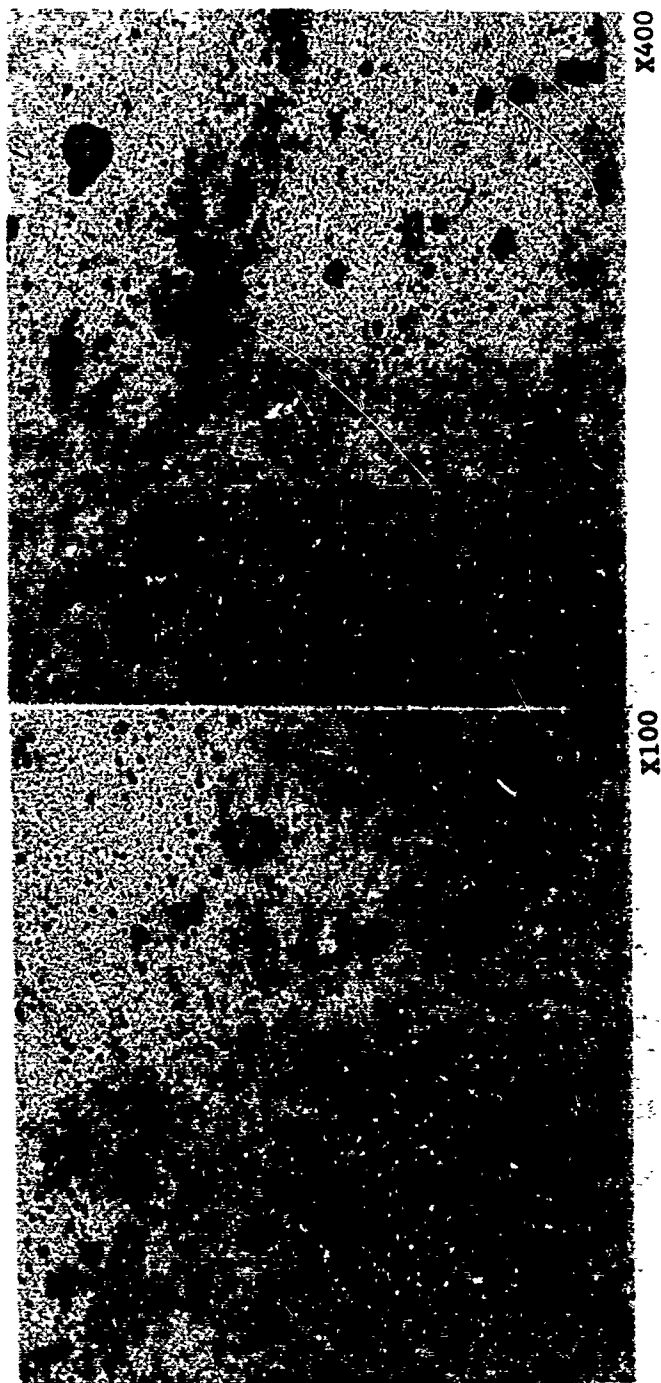


Figure A-13. Microstructures of 6061 Aluminum Solution-Treated at 980°F for 30 min, to T-4 Condition; Brazed in Air-Circulating Furnace, Resistance-Type; and Water-Quenched at 60°F

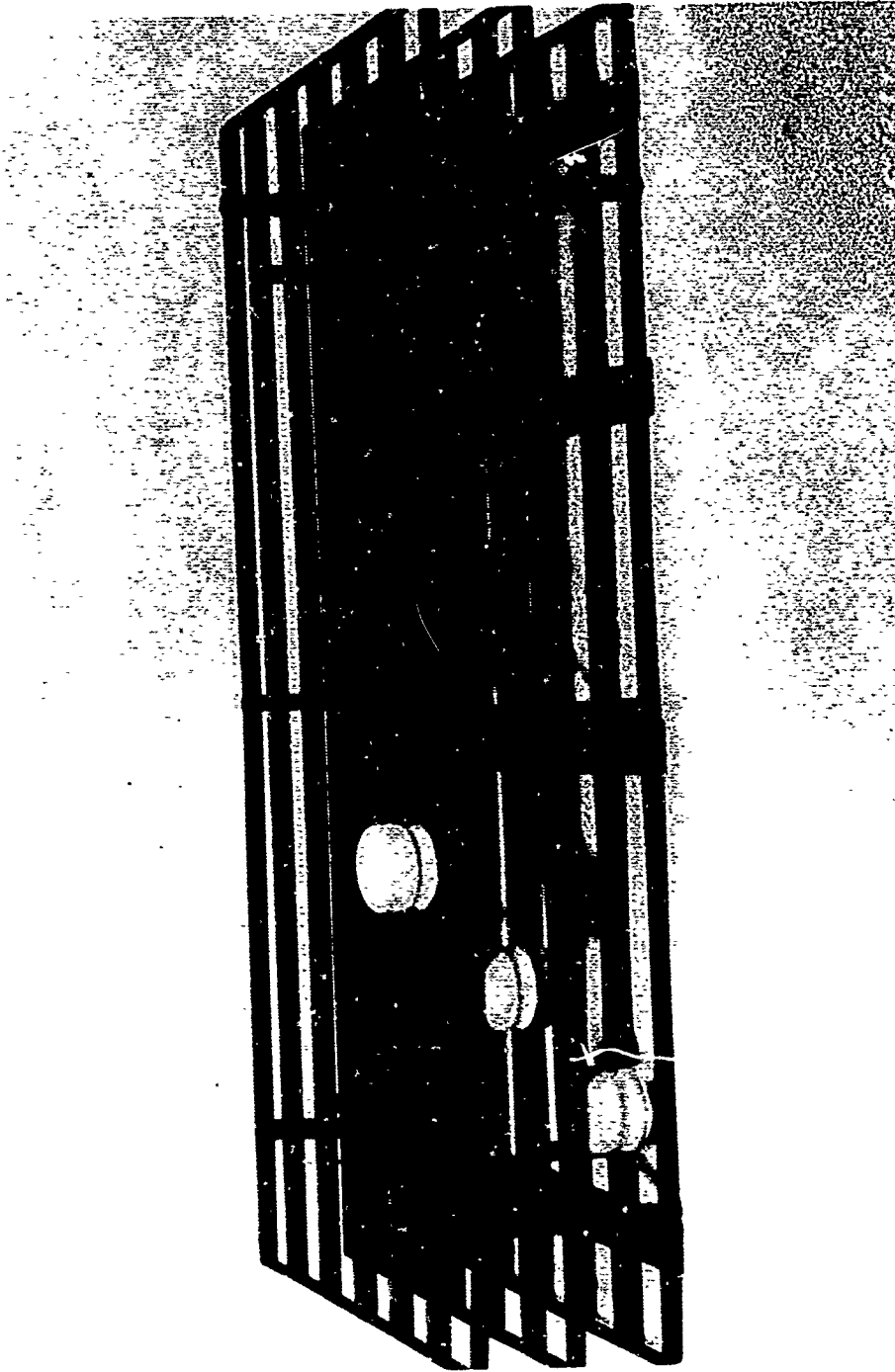


Figure A-14. Low-Cost Tooling for Vacuum Brazing-Gas Quenching
200 Bombulet Canisters at One Time

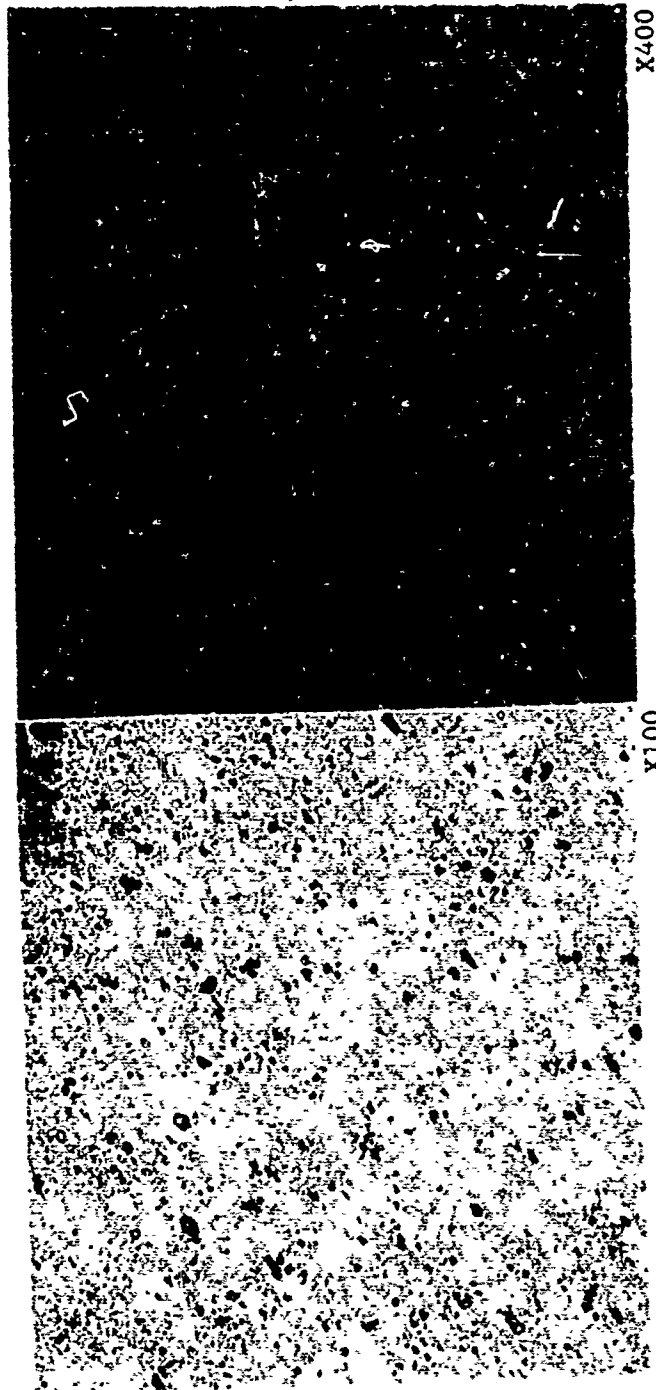


Figure A-15. Microstructures of 6061 Aluminum Aged at 300° to 320°F for 39 hr

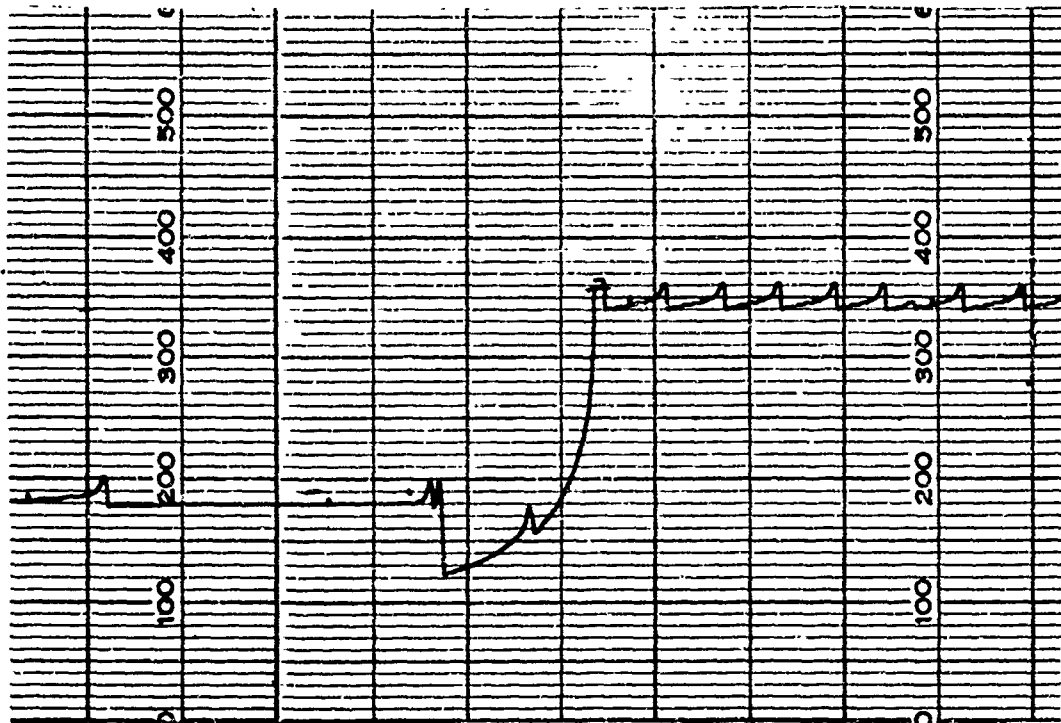


Figure A-16. Typical Duplex Aging Cycle: $350^{\circ} \pm 10^{\circ}\text{F}$ for 6 hr and $190^{\circ} \pm 10^{\circ}\text{F}$ for 48 hr

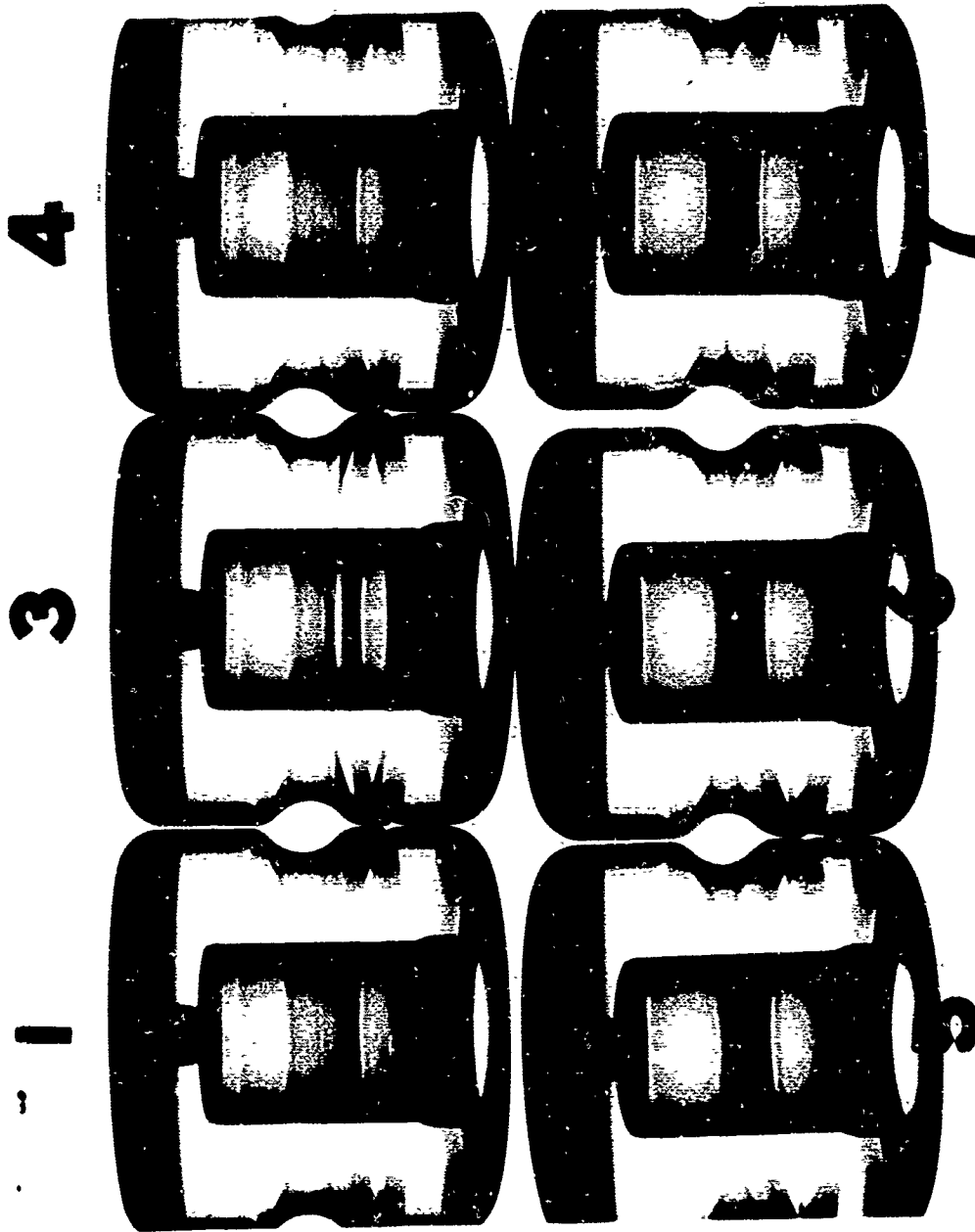


Figure A-17. Radiograph of Vacuum Brazed-Gas Quenched Bomblet Canisters

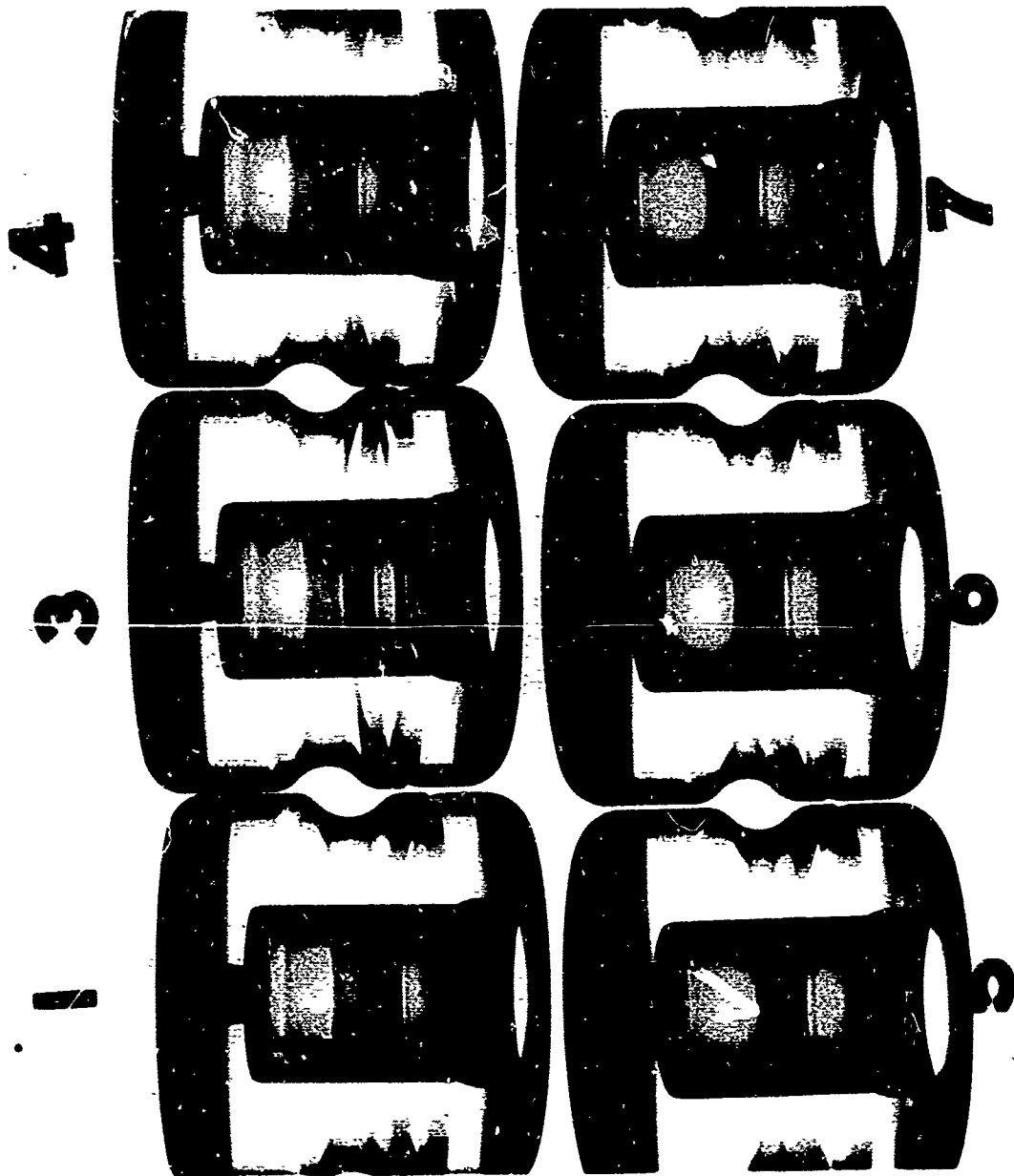


Figure A-18. Radiograph of Vacuum Brazed-Gas Quenched Bomblet Canisters

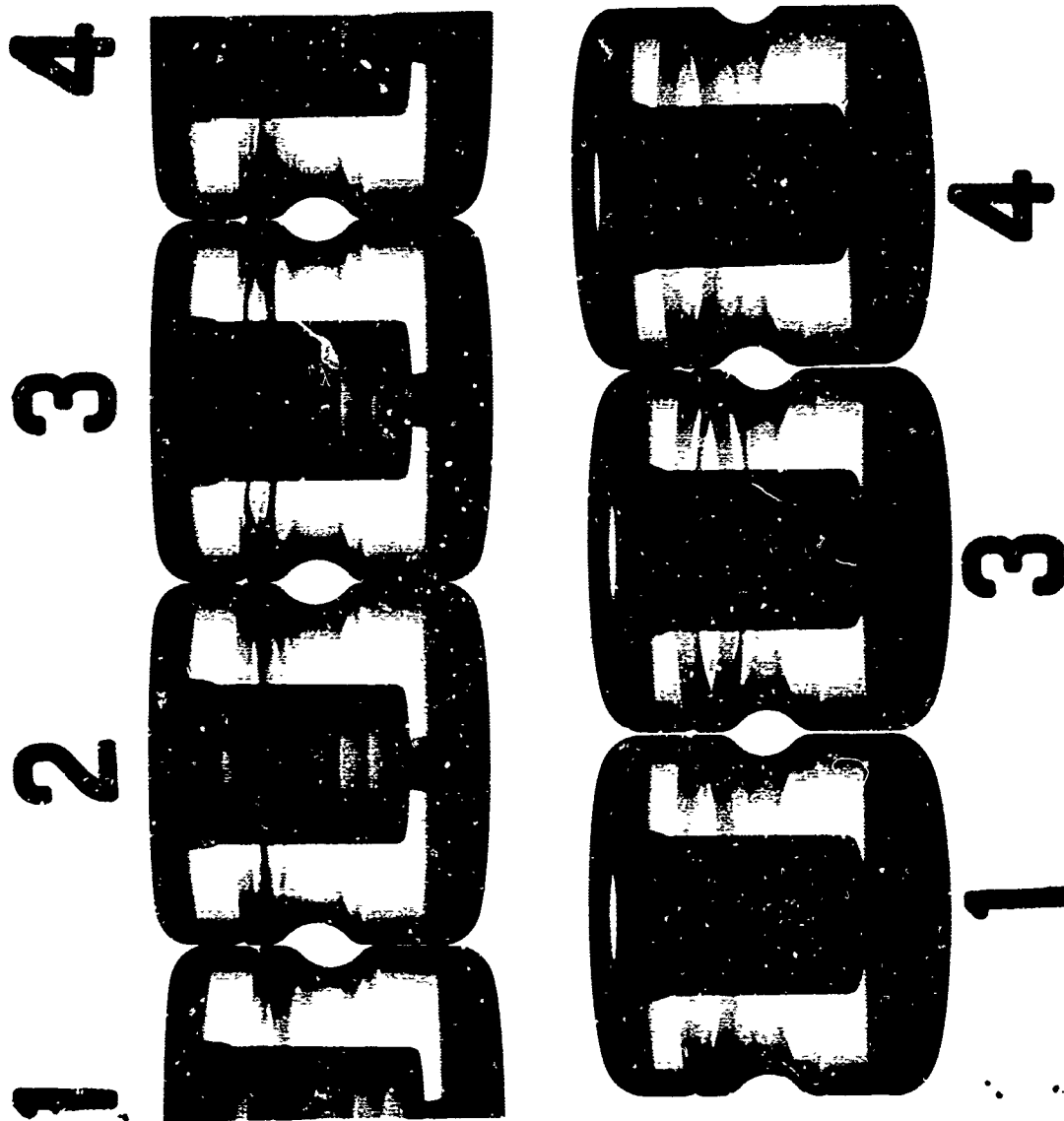


Figure A-19. Radiograph of Vacuum Brazed-Gas Quenched Bomblet Canisters

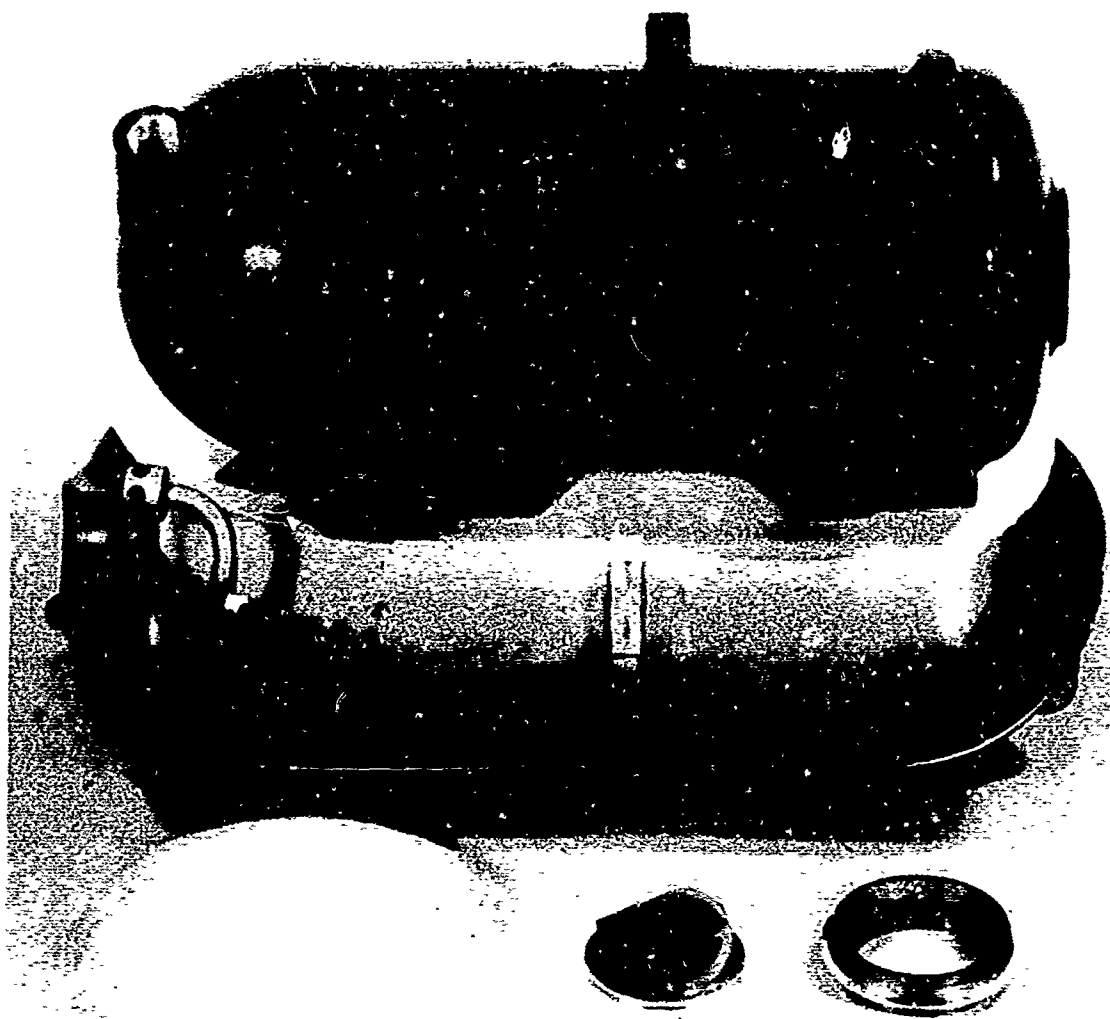


Figure A-20. Fuel Tanks, Future Applications for Vacuum
Brazing-Gas Quenching Process

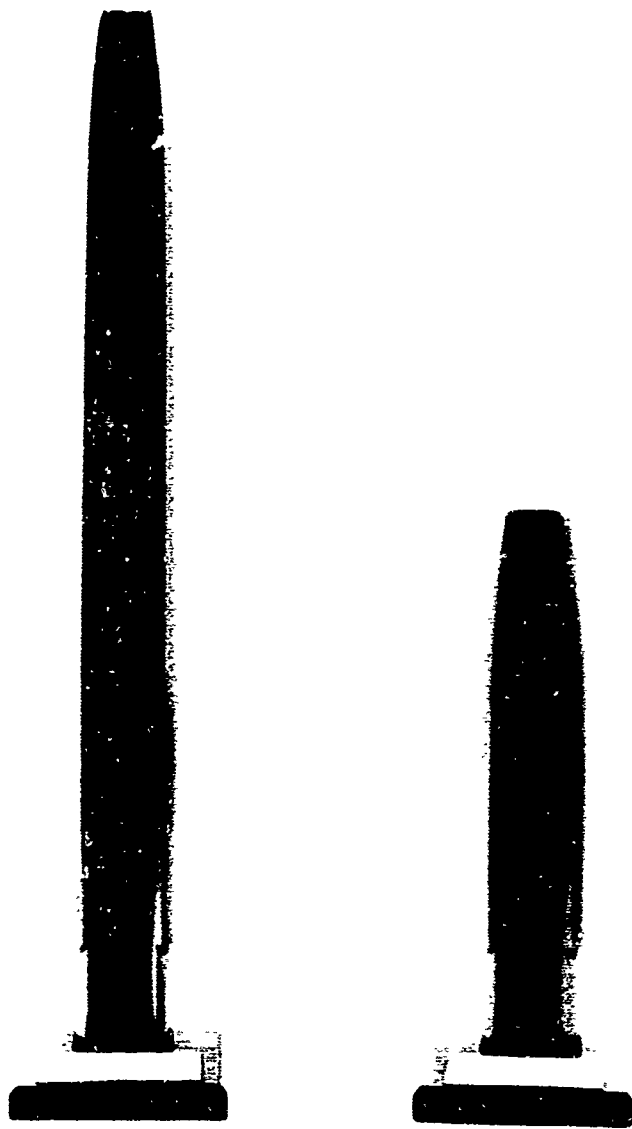
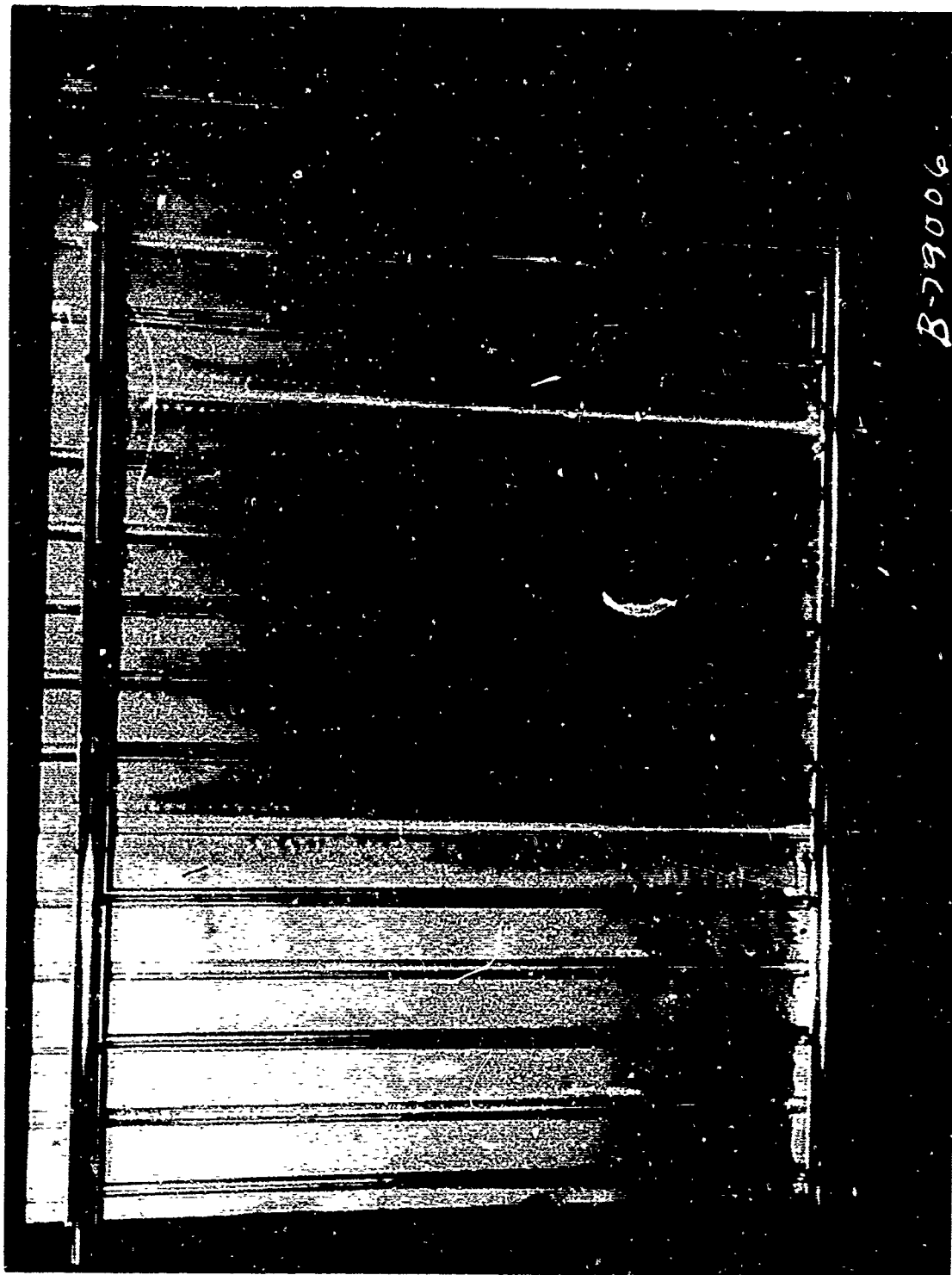


Figure A-21. Rocket Warheads, Future Applications for Vacuum Brazing-Gas Quenching Process



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Figure A-22. Other Applications of Fluxless Brazing

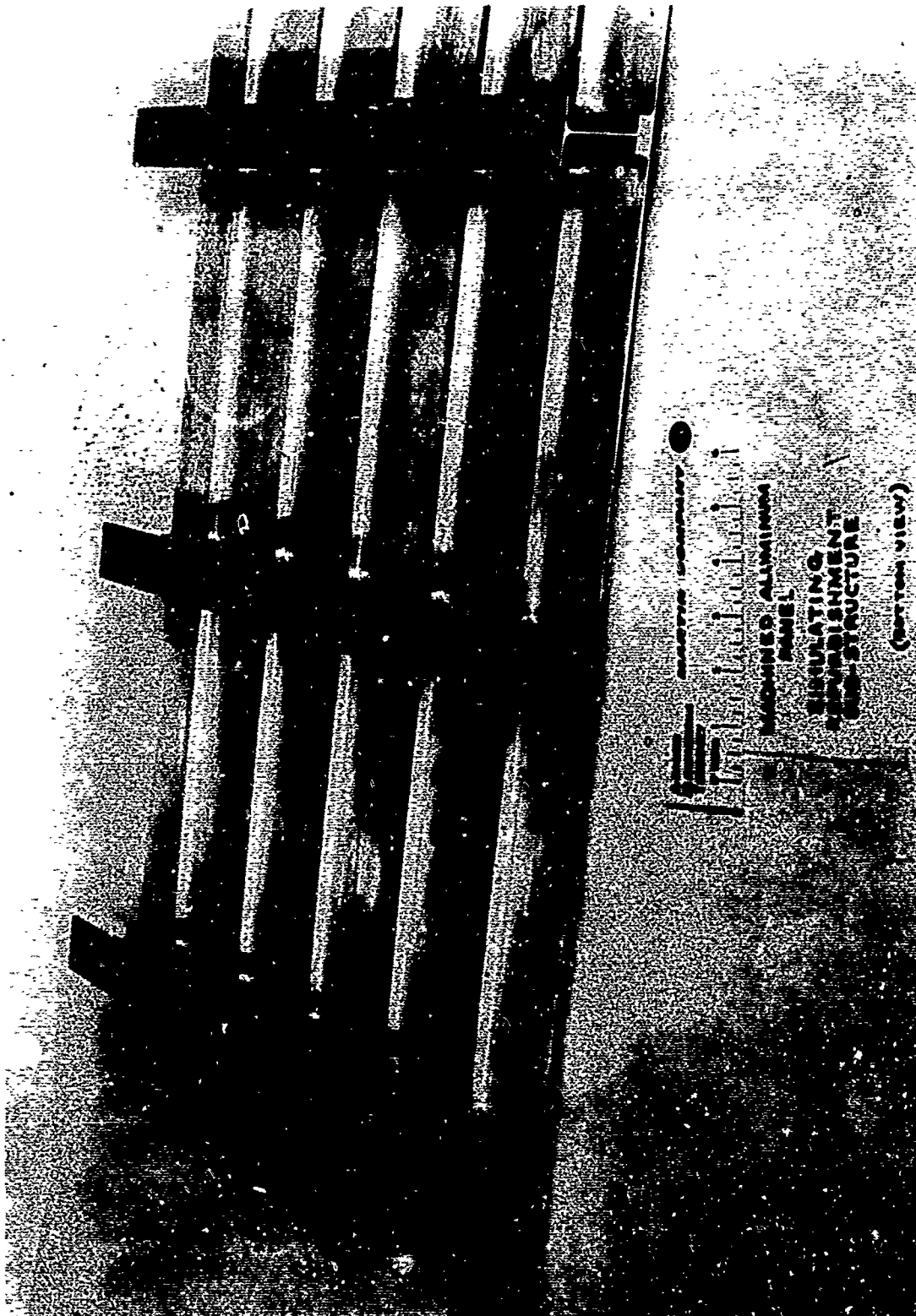


Figure A-23. Other Applications of Fluxless Brazing

Security Classification

(Security classification of title body of abstract and indexing annotation must be entered when the overall report is classified)

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